भारतीय मानक

Indian Standard

IS 18122 : 2023

जिबरेलिक एसिड, तकनीकी — विशिष्टि

Gibberellic Acid, Technical — Specification

ICS 65.100.10

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भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
मानक भवन, 9 बहादुर शाह ज़फर मार्ग, नई दिल्ली - 110002
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI - 110002
www.bis.gov.in www.standardsbis.in

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Price Group 6

Pesticides Sectional Committee, FAD 01

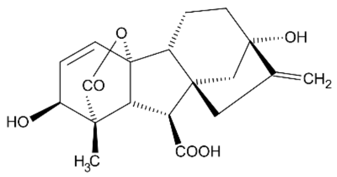
FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Pesticides Sectional Committee had been approved by the Food and Agriculture Divisional Council.

Gibberellic acid is a plant growth regulator agricultural use and Gibberellic Acid, technical used in the preparation of formulations of plant growth regulators.

Gibberellic acid is the accepted common name by the International Organization for Standardization for (3S,3aS,4S,4aS,7S,9aR,9bR,12S)-7, 12-dihydroxy-3-methyl-6methylene-2-oxoperhydro-4a, 7-methano-9b, 3 propenoazuleno[1,2-b] furan-4-carboxylic acid.

The empirical and structural formulae and the molecular mass of Gibberellic acid are given below:

Empirical Formula	*Molecular Formula*	*Molecular Mass*
$C_{19}H_{22}O_6$		346.37

In the preparation of this standard, due consideration has been given to the provisions of *Insecticides Act*, 1968 and the Rules framed thereunder. However, this standard is subject to the restrictions imposed under the *Insecticides Act* and Rules, wherever applicable.

The composition of the Committee responsible for the formulation of this standard is given in Annex C.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 2022 ‘Rules for rounding off numerical values (*second revision*)’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

GIBBERELLIC ACID, TECHNICAL — SPECIFICATION

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for gibberellic acid, technical.

2 REFERENCE

The following standards contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
IS 1070 : 1992	Reagent grade water — Specification (*third revision*)
IS 6940 : 1982	Methods of test for pesticides and their formulations (*first revision*)
IS 8190 (Part 1) : 1988	Requirements for packing of pesticides: Part 1 Solid pesticides (*second revision*)
IS 10946 : 1996	Method of sampling for technical grade pesticides (*first revision*)

3 REQUIREMENTS

3.1 Description

The material shall be in the form of white to off-white colour crystalline powder, free from extraneous matter.

3.2 The material shall also comply with the requirements specified in Table 1.

4 PACKING

The material shall be packed in suitable container and the packing material shall also confirm to IS 8190 (Part 1).

5 MARKING

5.1 The containers shall be securely closed and shall bear legibly and indelibly the following information:

a) Name of the material;
b) Name and address of the manufacturer;
c) Batch number;
d) Date of manufacture;
e) Date of expiry;
f) Net quantity;
g) Nominal gibberellic acid content, percent (*m/m*);
h) Cautionary notice as worded in the *Insecticides Act*, 1968, and Rules framed thereunder; and
j) Any other information required under the *Legal Metrology (Packaged Commodities) Rules*, 2011.

Table 1 Requirements of Gibberellic Acid, Technical
(*Clause* 3.2)

Sl No. (1)	**Characteristics** (2)	**Requirement** (3)	**Method of Test, Ref to** (4)
i)	Gibberellic acid content, percentage by mass, *Min*	90.0	Annex A
ii)	Melting point (°C)	223 to 226	IS 6940
iii)	*p*H in 1% aqueous medium	2.0 to 3.0	Annex B
iv)	Material insoluble in acetone, percentage by mass, *Max*	0.5	IS 6940

5.2 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

6 SAMPLING

Representative samples of material shall be drawn as prescribed in IS 10946.

7 TEST

7.1 Tests shall be carried out in accordance with the method referred in col **4** of Table 1.

7.2 Quality of Reagents

Unless specified otherwise, pure chemicals and reagent grade water (*see* IS 1070) shall be employed in the tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

[Table 1, *Sl No.* i)]

DETERMINATION OF GIBBERELLIC ACID CONTENT

A-1 PRINCIPLE

High Performance Liquid Chromatographic (HPLC) is used for the determination of gibberellic acid content.

A-2 APPARATUS

A-2.1 Analytical Balance

A-2.2 Volumetric Flask

A-2.3 HPLC

A-2.4 Chromatographic Parameters

Instrument: HPLC equipped with UV-VIS detector (*see* Fig. 1)

Column: Packing Material: ODS – C 18 (250 mm x 4.6 mm i.d, 5 µm) or equivalent

Mobile Phase: 0.5% Potassium Dihydrogen Phosphate: Acetonitrile (40 : 60), [*p*H 3.0 with Phosphoric acid]

Detection Wavelength: 208 nm

Temperature: Ambient

Injection Volume: 20 µl

Flow Rate: 1.5 ml/min

A-3 REAGENTS

A-3.1 Water – HPLC grade

A-3.2 Gibberellic Acid Reference Standards, of known purity

A-3.3 Phosphoric Acid – AR grade

A-3.4 Methanol – HPLC grade

A-3.5 Acetonitrile – HPLC grade

A-3.6 Potassium Dihydrogen Phosphate – AR grade

A-4 PROCEDURE

A-4.1 Prepration of Solvent Mixture

Make about 1 litre of 0.5% aqueous potassium dihydrogen phosphate. Mix it with acetonitrile with proportion of 40:60 (*v*/*v*). Adjust *p*H 3.0 with phosphoric acid. Filter and sonicate the solvent mixture. Homogenize the solvent mixture and allow it to attain room temperature.

A-4.2 Preparation of Standard Solution

Weigh accurately about 100 mg of Gibberellic Acid reference standard into a 100 ml Volumetric flask. Add about 10 ml of Methanol. Shake well and keep the solution for 30 minutes for intermittent shaking for complete dissolution. Make up the volume up to the mark with the solvent mixture.

A-4.3 Preparation of Sample Solution

Weigh accurately sufficient sample to contain about 100 mg of Gibberellic Acid in a 100 ml volumetric flask. Add about 10ml of Methanol. Shake well and keep the solution for 30 minutes for intermittent shaking for complete dissolution. Make up the volume up to the mark with the solvent mixture.

A-4.4 Estimation

Inject separately 20 µl each of the solution of standard and sample into HPLC unit. Inject the standard solution repeatedly, until the area of reference substance of two successive injections do not deviate from each other by more than 2 percent. Then inject the sample solution. The sequence of injection to be followed is as follows:
. .. C, S_1; C,S_2, c, . .

where

C = standard solution; and
S = sample solution.

Measure the peak areas of the reference standard and sample and compute the percentage of the Gibberellic Acid given as in **A-5**

A-5 CALCULATION

Gibberellic Acid content, percentage by mass = $\frac{A_2 \times M_1 \times P}{A_1 \times M_2}$

where

A_2 = Peak area of Gibberellic Acid sample solution;

M_1 = Mass of Gibberellic Acid reference standard;

P = Percentage purity of Gibberellic Acid reference standard;

A_1 = Peak area of Gibberellic Acid reference standard; and

M_2 = Mass of Gibberellic Acid sample.

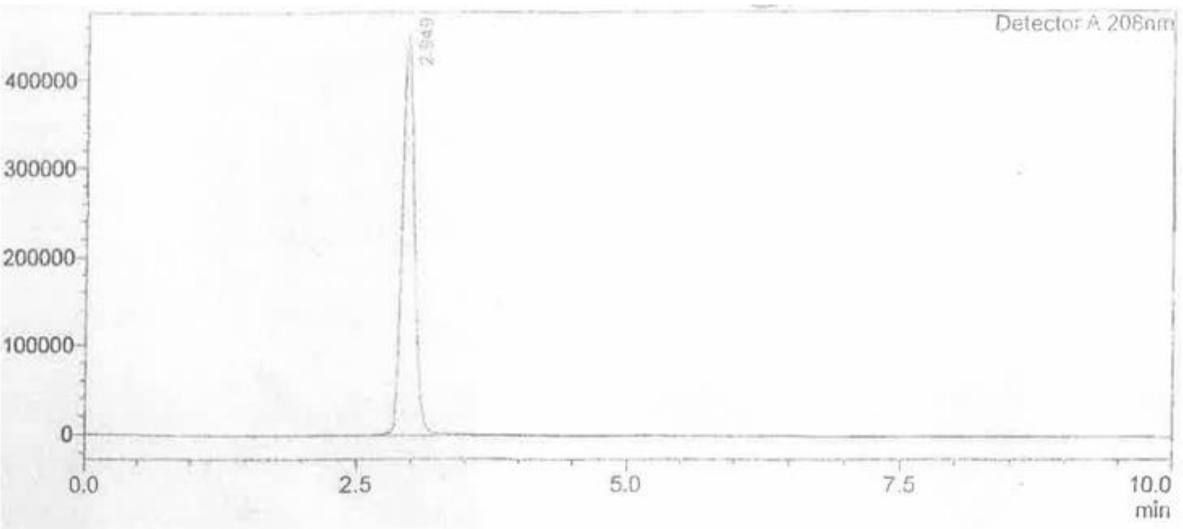


FIG. 1 CHROMATOGRAM OF GIBBERELLIC ACID, TECHNICAL

ANNEX B

[*Table* 1, *Sl. No.* iii)]

DETERMINATION OF *p*H VALUE

B-1 OUTLINE OF METHOD

The *p*H value of a mixture of sample with water is determined by means of *p*H meter and an electrode system.

B-2 REAGENTS

B-2.1 Di-Sodium Tetraborate Buffer Solution

0.05 mol/l, commercially available or self-prepared solution (*see* Note 1).

B-2.2 Potassium Hydrogen Phthalate Buffer Solution

0.05 mol/l, commercially available or self-prepared solution (*see* Note 2).

B-2.3 Water

Distilled or de-ionized, in equilibrium with CO_2 from the air (*see* Note 3).

B-3 APPARATUS

B-3.1 *p*H Meter

Capable of at least two points calibration.

B-3.2 Electrode System

Glass electrode system, conditioned and stored according to the manufacturer’s instructions.

B-3.3 Measuring Cylinder Stoppered, 100 ml.

B-4 PROCEDURE

B-4.1 Calibration

Operate the *p*H meter and the electrode system according to the manufacturer’s instructions. Calibrate the measuring system (*p*H meter and electrode) according to the manufacturer’s instructions using at least two appropriate buffer solution.

B-4.2 Measurement of *p*H Value

B-4.2.1 *Measurement of Diluted Sample*

Weigh 1.0 g of sample (*see* Note 4) into a measuring cylinder containing about 50 ml water (*see* Note 5), make up to 100 ml with water and shake vigorously until completely mixed or dispersed. If necessary, transfer the solution or dispersion to beaker (200 ml) and allow any suspended material to settle for 1 min. Ensure that the temperature of the sample/water mixture does not differ from the temperature of the liquid and immediately start the stopwatch (*see* Note 7). Record the *p*H value after 1 and 2 minutes, without stirring during the measurement. If the two *p*H values differ by more than 0.1 *p*H units, record and reports the *p*H value minutes after immersing the electrode.

B-4.2.2 *Measuring of Undiluted Aqueous Formulation*

Transfer enough sample to a beaker (100 ml). Ensure that the temperature of the sample/water mixture does not differ from the temperature of the liquid and immediately start the stopwatch (*see* Note 7). Record the *p*H value after 1 minute and 2 minutes, without stirring during the measurement. If the two *p*H value differ by more than 0.1 *p*H units, record and reports the *p*H value minutes after immersing the electrode.

B-4.3 Reporting of Results

Give the result to the nearest 0.1 *p*H unit and report the following conditions:

B-4.3.1 The concentration of the mixture that has been measured (normally 1% *w*/*v*, or 1% *v*/*v* for liquid samples).

B-4.3.2 Whether or not the *p*H has been measured in an undiluted sample.

B-4.3.3 The type of water that has been used.

B-4.3.4 The temperature at which the *p*H has been measured.

Notes

1 Di-sodium tetraborate solution, 0.05 M (*p*H see below); and
Dissolve 19.07 g di-sodium tetraborate ($Na_2B_4O_7.10H_2O$) in water (2.2) and make it up to 1 000 ml. Do not keep the solution for longer than one month.

*p*H values:

Temperature (℃)	10	15	20	25	30
pH	9.29	9.26	9.22	9.18	9.14

2 Potassium hydrogen phthalate solution, 0.05 M (*p*H 2.0 and 7.0):
Dissolve 10.21 g potassium hydrogen phthalate (HOOC-C_6H_4-COOK) in water and make up to 1 000 ml. The temperature coefficient of the *p*H of this buffer solution can be neglected between 10 and 30 ºC. Do not keep the solution for longer than one month.

3 If necessary, the water used should be freshly boiled and cooled to room temperature.

4 In case of liquid samples which are not too viscous, e.g. emulsion concentrates, 1 ml (or more) of the sample may be used.

5 Special types of water instead of distilled or de-ionized water may be prescribed.

6 During the measurement of *p*H values of samples a randomly fluctuating reading of the *p*H meter may be observed. The reason is normally that the concentration of the ions in the sample (diluted or undiluted) is too low, or because of an interaction between the particles or droplets in suspension with the electrode. If the ion concentration is too low, some drops of concentrated sodium chloride solution may be added to stabilize the reading.

7 When using an automated *p*H meter, where the measurement stops automatically as soon as the *p*H changes are less than a pre-set drift value of 0.1 *p*H units/min, a measurement period less than 10 min is acceptable and the resulting value should be reported.

ANNEX C

(*Foreword)*

COMMITTEE COMPOSITION

Pesticides Sectional Committee, FAD 01

Organization	*Representative(s)*
Directorate of Plant Protection Quarantine and Storage, Faridabad	Dr Ravi Prakash (***Chairperson***)
All India Biotech Association, New Delhi	Shri Saurabh Singhal Shri Shah Ji Dhar (*Alternate*)
Central Insecticide Board and Registration Committee, Faridabad	Secretary Dr Vandana Seth (*Alternate*)
Central Insecticide Laboratory, Faridabad	Dr Archana Sinha Shri Subhash Chaudhary (*Alternate*)
Consumer Guidance Society of India, Mumbai	Shri Sitaram Dixit Dr M. S. Kamath (*Alternate*)
Crop Care Federation of India, New Delhi	Dr J. C. Majumdar
Crop Life India, New Delhi	Shri Asitava Sen Ms Nirupama Sharma (*Alternate*)
CSIR-Indian Institute of Toxicology Research, Lucknow	Director Dr Sheelendra P. Singh
FMC India Pvt. Ltd., Bengaluru	Shri Chirag Patel
Food Safety and Standards Authority of India, New Delhi	Advisor (Standards)
IDMA Laboratories Ltd., Chandigarh	Dr Indra Rai
Indian Agricultural Research Institute, New Delhi	Director
Indian Institute of Packaging, Mumbai	Dr Tanweer Alam
Indian Pest Control Association, New Delhi	Shri Udayan Ghosh
Institute of Pesticide Formulation Technology, Gurgaon	Dr M. Vairamani
Ministry of Agriculture, Department of Agriculture, Chennai	Joint Director of Agriculture (*RES.*) Deputy Director Lab (*Alternate*)
National Centre for Integrated Pest Management, New Delhi	Dr Sumitra Arora

Organization	*Representative(s)*
National Institute of Plant Health Management, Hyderabad	Dr Mahesh Saini Ms T. Sridevi (*Alternate*)
Pesticide Manufactures and Formulators Association of India (PMFAI), Mumbai	Dr Archana Srivastava Dr Uday Kumar (*Alternate*)
Regional Pesticides Testing Laboratory, Chandigarh	Shri V. Vasu
In Personal Capacity (*4-6-90/2/8/2, Sri Devi Nilayam Tejaswinagar Coloni, Attapur, Hyderabad – 500048*)	Shri C. V. Rao
In Personal Capacity (*263, Sector 28, Faridabad – 121008, Haryana*)	Shri Vipin Saini
BIS Directorate General	Shrimati Suneeti Toteja, Scientist 'E'/ Director And Head (Food and Agriculture) [REPRESENTING DIRECTOR GENERAL (*Ex- officio*)]

Member Secretary
Shri Kuldeep Mittal
SCIENTIST 'B'/Assistant Director
(Food and Agriculture), BIS

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Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones: 2323 0131, 2323 3375, 2323 9402 *Website*: www.bis.gov.in

Regional Offices:		*Telephones*
Central	: 601/A, Konnectus Tower -1, 6th Floor, DMRC Building, Bhavbhuti Marg, New Delhi 110002	2323 7617
Eastern	: 8th Floor, Plot No 7/7 & 7/8, CP Block, Sector V, Salt Lake, Kolkata, West Bengal 700091	2367 0012 2320 9474
Northern	: Plot No. 4-A, Sector 27-B, Madhya Marg, Chandigarh 160019	265 9930
Southern	: C.I.T. Campus, IV Cross Road, Taramani, Chennai 600113	2254 1442 2254 1216
Western	: Plot No. E-9, Road No.-8, MIDC, Andheri (East), Mumbai 400093	2821 8093

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